E113-1105

LEVEL II

FABRICATION OF FIRE RESISTANT TRANSPARENCIES

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ABSTRACT

NASA/Ames Research Center in the early 70's, as part of its work on development of fire resistant aircraft materials, produced a transparent epoxy compound with considerably more fire resistance than conventional plastic transparencies. This compound, identified as EX-112, is a combination of Shell Epon 825 resin and Callery Chemical Co. trimethylboroxine as the hardening agent. The EX-112 can be cast into sheets and laminated to another plastic transparency, such as polycarbonate, to result in a tough, fire resistant composite transparency. Alternatively the epoxy can be cast directly onto the other plastic material.

Hughes Aircraft Company was given the task to produce a limited number of typical aircraft transparencies. The techniques which were developed included methods of casting sheets, curving the sheets into the required contours and then laminating the epoxy transparencies to other plastics. Boeing 737 laminated side windows were made, using the epoxy as the outer surface and polycarbonate as the inner surface. Identical sized windows were also made using a single cast monolithic sheet of EX-112.

One foot square laminated samples for ballistics tests were made with polycarbonate and acrylic substrates.

An acrylic A4-D canopy was coated with approximately 1/8 inch of EX-112 using a technique whereby the epoxy was cast directly onto the acrylic.

Tests of the composite transparencies consisted of determination of heat distortion temperature, limiting oxygen index, burning rate, thermal conductivity and the coefficient of thermal expansion.

The results indicated that a substantial improvement in fire resistance could be obtained over state-of-the art acrylic transparencies.

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Describes methods of casting sheets, curving the sheets into the required contours and then laminating the epoxy transparencies to other plastics. Laminated side windows were made with epoxy as the outer surface and polycarbonate as the inner surface. Identical sized windows were also made using a single cast monolithic sheet of EX-112.

Tests of the composite transparencies consisted of ballistics tests, determination of heat distortion temperature, limiting oxygen index, buring rate, thermal conductivity and the coefficient of thermal expansion.

Results indicated a substantial improvement in fire resistance over state-of-the art acrylic transparencies.

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INTRODUCTION

A number of investigations are currently being made to improve plastic aircraft windows and airplane canopies from the standpoint of fire resistance. Such windows are lighter than glass, considerably more shatterproof and could be fabricated of materials which would have high flame resistance, by virtue of char formation on exposure to high heat.

One concept for making plastic windows is to cast a monolithic window using a transparent, char forming, epoxy resin. Another concept is to laminate a very tough, strong transparent polycarbonate sheet with the transparent char forming epoxy sheet, using a compatible interlayer material.

To investigate the feasibility of these two concepts, Hughes Aircraft Company was selected by NASA/Ames Research Center to fabricate a number of windows of each type similar in size and contour to the Boeing 737 outer window (Part No. 65-45791). An epoxy formulation, EX-112, developed by NASA Ames Research Center, was utilized in the fabrication of both types of windows. An ethylene terpolymer (ETP) interlayer developed by Monsanto was used to laminate the epoxy to the polycarbonate. In addition to the 737 type windows, two types of composite ballistic transparencies were made, and an A4-D acrylic canopy was coated with EX-112 material.

The determination of the flame resistance and other thermal characteristics of the various transparencies was done by a series of tests performed by NASA/Ames. These included heat distortion temperature, limiting oxygen index, burning rate, thermal conductivity and coefficient of thermal expansion. The ballistic behavior was determined by the Naval Surface Weapons Center, Dahlgren, Virginia. The projectiles used for testing were 0.30 armor piercing and ball rounds and 0.22 caliber fragment simulating rounds.

II. SUMMARY AND CONCLUSIONS

This paper describes the development of the processing technique used in the fabrication of transparent, heat-resistant aircraft windows of the Boeing 737 configuration. Several types of transparent composite ballistic samples were made, and an A4-D canopy was coated with an epoxy overlay.

Two types of windows were fabricated:

1) monolithic type, one-half inch thick, made from NASA epoxy formulation EX-112 (Epon 825 containing 5 PHR of trimethoxyboroxine), and 2) a laminated type consisting of a quarter-inch thick polycarbonate inner ply bonded to a quarter-inch thick EX-112 ply with an ethylene terpolymer interlayer material.

By the proper choice of processing conditions, the feasibility of fabricating heat-resistant aircraft windows of the above types has been clearly demonstrated. The principal difficulties involved the development of conditions for the casting and curving of the relatively large epoxy sheets required. Completely curing these epoxy sheets in the casting mold was found to be impractical due to curing shrinkage and resultant cracking. Additionally, the curving of the completely cured sheets to the desired radius of curvature was difficult to control. Both problems were solved by partially curing the epoxy in the casting fixture and then completing the cure and curving to the required radius of curvature in a single operation. The laminating operation was accomplished readily in an autoclave which was modified to allow its operation as a vacuum chamber. No difficulty was experienced in the machining of the monolithic or laminated blanks to the final 737 window configuration.

The ballistic samples were made by laminating some with an interlayer, and also by casting the epoxy against the acrylic. The A4-D canopy was coated with EX-112 by casting the epoxy directly onto the acrylic. Both processes could be used in production.

III. DETAILED PROCEDURES

Essentially the fabrication process for production of the 737 windows consisted of six steps:

- Tooling preparation
- Epoxy sheet casting
- Curving and final curing of the epoxy ply
- Polycarbonate curving
- Lamination
- Final Finishing

A. Tooling Preparation

Initial tests were made by casting the epoxy between quarter-inch thick glass plates covered with a sprayed PVA parting film and/or silicone release agent and wax. Only partial success was obtained, since the optical surface was poor, and release was not perfect. Mylar film worked reasonably well as a parting film, except that some "orange peeling" was observed in the epoxy castings, apparently due to curing shrinkage.

Far better results were obtained by the use of half-inch thick aluminum tooling plates covered with stainless steel Ferrotype plates (bonded with American Cyanamide FM 123-2 film adhesive). Fair to good release was obtained by coating the Ferrotype plates with Ram Chemical Co. 87X76 mold release followed by Simoniz or carnauba wax. The fixtures used to make the 737 windows were 18x22 in. The plates were clamped together with spring clamps, or bolts, using a Dow Corning DC-4 coated neoprene or silicone gasket and aluminum spacers.

B. Casting

The casting formulation, originally developed by NASA-Ames, Ref. 1, consisted of the following (the amount shown is sufficient to make one 16"x20"x1/4" ply):

Shell Chemical Co. Epon 825	-	1200 gm
Callery Chemical Co. Trimethylboroxine	-	60 gm
Patent Chem. Co. Perox blue dye (.17 gm in 125 gm Epon 828)	-	50 drops

The dye-epoxy mixture is heated to 50±3°C (120±5°F). Then the TMB is added and mixed rapidly and then the mixture immediately vacuum degassed. As soon as the mixture "breaks" in the vacuum chamber it is removed and immediately cast through a paint strainer into the mold which has been preheated to 50±3°C (120±5°F). The temperature is then raised to 71±3°C (160±5°F) as rapidly as possible, and kept at 71°C for two hours.

C. Curving and Final Cure

At the end of the two hour "semi-cure" period the mold is opened, while still hot, and the "rubbery" casting is immediately removed and placed on a Ferrotype covered bend fixture. (The Ferrotype is bonded to a curved aluminum plate and parting agent coated, similar to the casting forms).

The entire assembly is placed in a nylon film bag (Allied Chemical Co. Capran) which has fittings for argon flushing.

A final cure, with slow argon flushing, is as follows:

3 hours at 135±3°C (275±5°F)
4 hours at 168±3°C (335±5°F)

The assembly is then slowly returned to room temperature. The casting is examined, trimmed to 13x17 in. and buffed, if required.

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D. Polycarbonate Curving

The curving of the 13x17x1/4 inch polycarbonate panels is done using the same tooling employed for curving and curing of the epoxy plies.

Prior to the curving operation the flat polycarbonate sheets are circulating air oven dried for 24 hours at 130$\pm$3°C (265$\pm$5°F), or 96 hours at 115$\pm$3°C (220$\pm$5°F). The latter procedure is used for week-end drying. After drying, the panels are kept, with a desiccant, in sealed polythylene bags, unless they are to be used immediately.

To curve the polycarbonate it is placed on the Ferrotype form and held down with five spring clamps, and 1x18x1/4 in. aluminum bars at each end. One clamp and a small bar are also placed in the middle, at each side. Two #36 wire thermocouples are placed, one at each end of the plastic sheet, one under and one on top of the sheets. Aluminum tape is used to hold the couples. Aluminum foil is placed over the entire assembly to act as a dust shield.

The oven is set to a maximum temperature of 157°$\pm$3°C (315$\pm$ 5°F), and the part temperature is monitored by a recorder. When the plastic reaches 155$\pm$3°C (310$\pm$5°F), the oven temperature is reduced so the part can be kept at 155°C for 10 minutes, after which the oven is turned off and the doors are opened. The part is allowed to cool to room temperature, before removal from the fixture. During the heating period, the assembly should be checked for buckling, due to differential expansion. If this is found,the clamps should be loosened from one end, the sheet smoothed down and reclamped. A slight over or under bend (1/8 in. max.) is not deleterious.

E. Lamination

Prior to lamination, the epoxy and polycarbonate plies are rinsed with detergent solution and alcohol until a water break-free surface is obtained. The terpolymer is likewise washed with two alcohol rinses. All the components are then dried for a minimum of 4 hours at 50$\pm$3°C (120$\pm$5°F) in a vacuum oven.

The laminate is then assembled as shown in Fig. 1. The polycarbonate sheet is laid on a Ferrotype, followed by the polyethylene terpolymer. Care should be taken to entrap a minimum number of air bubbles under the ETP. Any visible bubbles should be carefully pricked and smoothed out by hand, to avoid bubbles in the final laminate.

After removal of as many bubbles as possible under the terpolymer, the epoxy ply should be carefully placed, starting at one end, so as to also minimize air entrapment. Three or four fine wire thermocouples are fastened to corners of the laminate using aluminum pressure sensitive tape. The laminate assembly is then wrapped with two layers of Burlington #51789 nylon fabric, followed by four layers of 1/8 in. thick SAE STD. F-6 felt. The entire package is then oven dried fo 16 hours minimum at 50±3°C (120±5°F) in a vacuum oven.

After the drying period, the package is installed on a curved fixture covered by a 4 mil Capran vacuum bag, Two sealants were found best for holding the vacuum bag to the fixture. A low temperature sealer (L.T.Fuller-O'Brien 3992) is placed on the outer periphery of the bag, and inside a high temperature sealant, Shnee and Morehead #9156, is used. Two sealants are used since the low temperature resistant,highly tacky material establishes the initial seal and enables the vacuum bag to be drawn down fully, thus also compressing the high temperature resistant seal. On reaching the higher temperatures, the latter material is then responsible for maintenance of a good seal around the bag.

The vacuum bag is checked for maintenance of a satisfactory vacuum, then the entire assembly-laminate package and fixture is placed in the vacuum chamber. Thermocouple and electrical leads are fastened to appropriate lead-throughs in the chamber wall.

Since it is important that the rate of rise of temperature during the laminating operation follow a definite curve, the laminating fixture has its own electrical heating blanket. The rate of temperature increase is controlled from outside the chamber by a large Variac. The heating rate is monitored by means of the three thermocouples on the part, and two others on the laminating fixture.

In order to accurately determine the pressure inside the bag, two vacuum outlets are used, one at each end of the bag, one going to the vacuum pump, and the other to the bag manometer, or pressure gage. A second manometer is installed with one leg connected to the bag and the other to the vacuum chamber interior; thus this manometer will continously indicate the differential pressure between the bag and the vacuum chamber.

Prior to turning on the heat the bag is evacuated to at least 29-1/2 in. Hg. Then the chamber is evacuated until the chamber pressure is approximately 2 in. Hg above the bag. The fixture heater is then turned on and a heating rate of approximately 42°C (75°F) per hour is used. The temperature rise and pressure differential are maintained as shown in Fig. 2, Ref. 2. After one hour at 127°C (260°F) the heat is turned off, the chamber

is allowed to reach full atmospheric pressure, and after cooling to below 40°C (104°F), the vacuum bag pressure is released and the laminate is removed. A final trimming and buffing then completes the window.

Ballistic Samples

Laminated samples were made for ballistic tests by laminating 1/8 inch thick cured EX-112 flat sheets to 1/4 acrylic and polycarbonate sheets, using the ETP interlayer and the same laminating procedure as above.

Another type of ballistic sample was made in which the EX-112 was cast directly onto an acrylic ply. By maintaining the composite transparency in the casting fixture, it was possible to obtain a complete cure in the epoxy. Differential thermal expansion strains were apparently not severe enough to cause any delamination on cooling.

A4-D Canopy Coating

Using the experience gained in the above operations, it was possible to coat an entire A4-D airplane canopy. In order to cast the coating directly onto the acrylic, a special fixture was made which, with the canopy held in an inverted position, was molded to the canopy lines, with an allowance of 1/8 to 1/4 inch. The tool was coated with Garalease prior to casting.

Casting was done with the tool and canopy in an oven at 71°C (160°F). The assembly was held for four hours at 71°C after pouring, after which it was slowly cooled (overnight). The coated canopy was removed the next day. An extra long post-cure (120 hours) up to a maximum temperature of 220°F was used to bring the epoxy up to its final hardness of Barcol 30-35.

IV TESTING

The Boeing 737 windows fabricated from EX-112 were evaluated for fire protection performance using a stretched polymethylmethacrylate 737 window as the control. An oil burner that provided a heat flux of 11.3×10^4 w/m^2 was used to simulate a JP-4 fuel fire. The acrylate window exposed to this environment exhibited the typical melting with combustion; burn-through occured in about 1 minute. The EX-112 prototype exposed to the same environment formed a hard, tough, surface char, which maintained internal protection for this window for about 6 minutes or about six times that of the standard window. Burn-through occured from thermochemical failure due to a small amount of stress.

Other thermophysical and flammability properties are summarized in Table I (Reference 1) and are compared to the acrylic currently used as the other aircraft window. The superior thermal performance of the EX-112 is exhibited by its higher limiting oxygen index, higher heat distortion temperature and longer time to burn-through. Since the major use intended for these transparencies is to provide thermal protection beyond that afforded by current windows and canopies, the most critical test was exposure to the heat flux of a JP-4 fuel proof fire. To simulate this this exposure, the samples were tested in the Ames T-3 thermal test facility. This facility is capable of simulating the typical fluxes encountered in large scale aircraft crash fires. As shown in Table I (Ref. 1) the acrylic transparencies exposed to this environment burn through in approximately 1-1/2 minutes, while the EX-112 epoxy exposed to the same conditions had not burnt through after 16 minutes. Equally as good in this respect is the laminated versions of this material, even when when laminated to acrylic substrates.

In regard to the epoxy systems for military aircraft canopies, the principal test was ballistic tolerance. The monolithic epoxy system is quite brittle and only laminated systems were ballistically tested. Epoxy cast directly on acrylic suffered delamination under ballistic impact as one would expect. However, epoxy laminated with an interlayer adhesive to either acrylic or polycarbonate substrates performed well, and had a ballistic tolerance that in some cases exceeded that of the stretched acrylic panels.

Acknowledgment

The development of the processing methods and fabrication techniques described herein was performed under NASA Contract NAS2-7765.

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References

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FIGURE 1 LAMINATION PACKAGE

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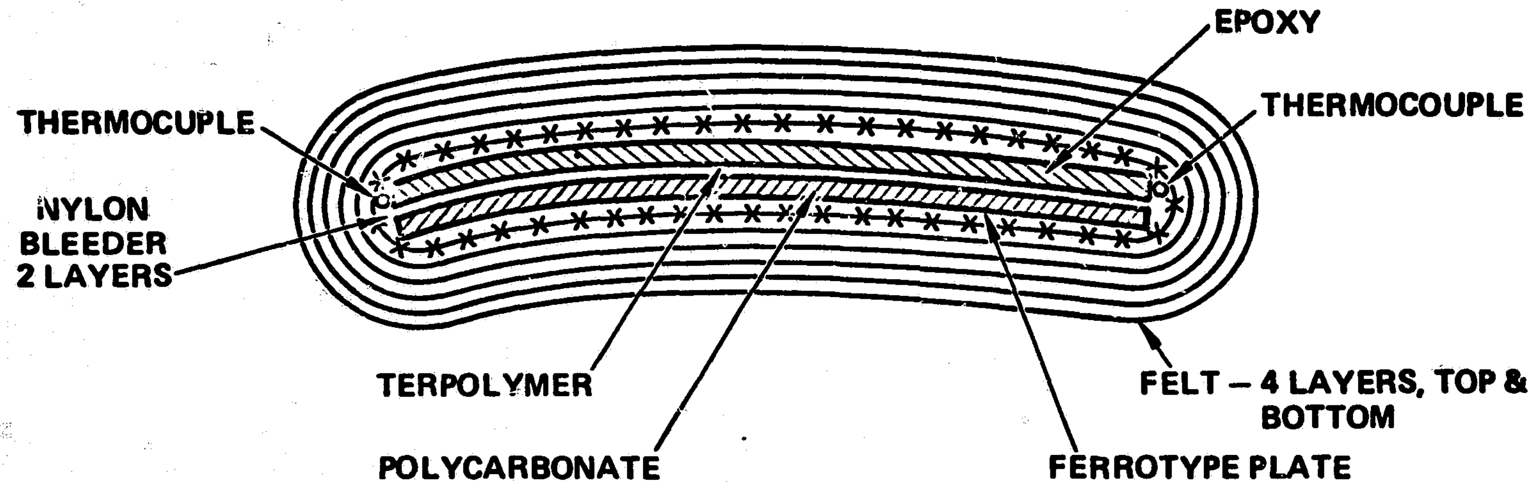

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FIGURE 2 LAMINATION CURE AND PRESSURIZATION CURVE

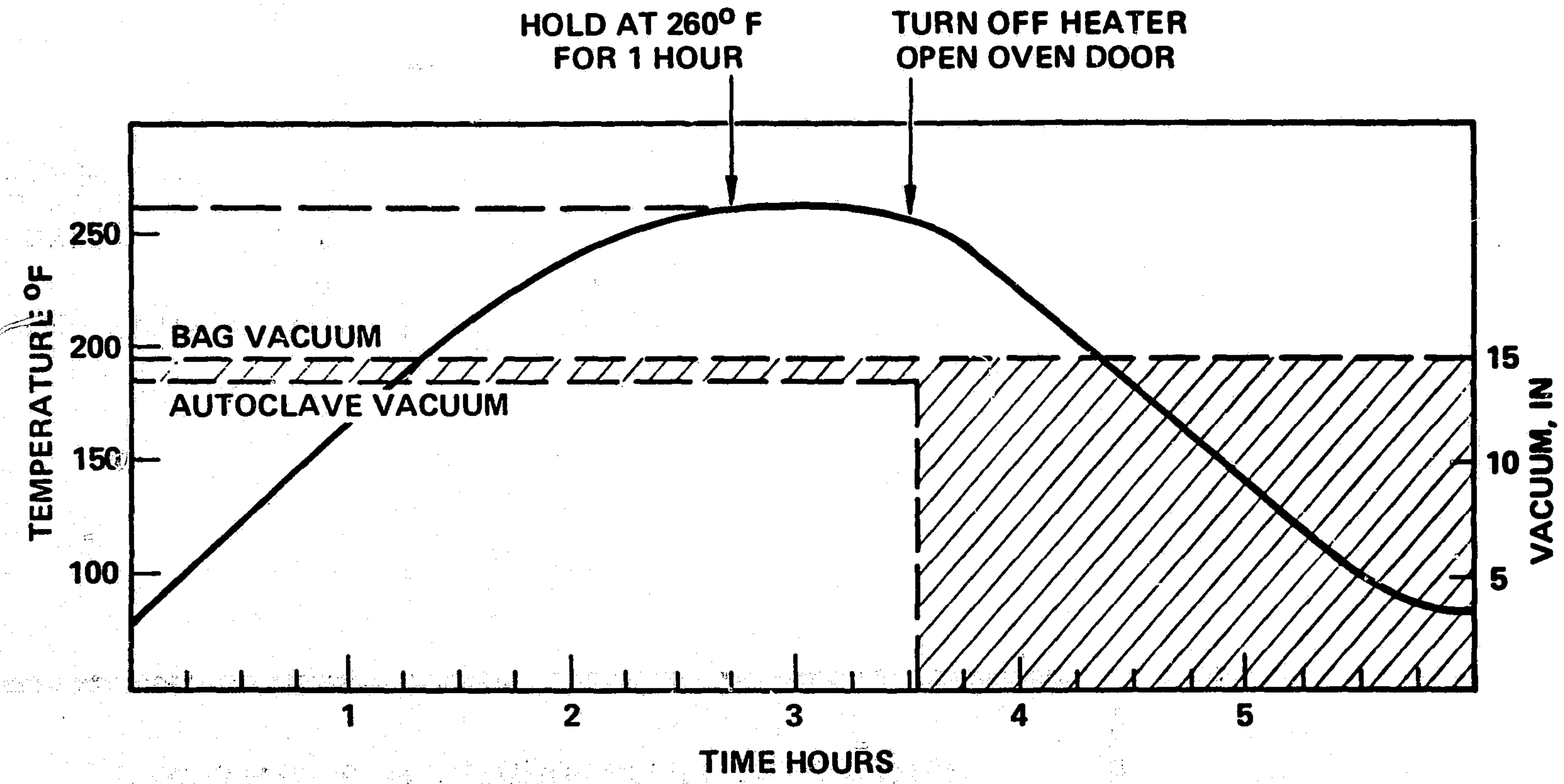

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TABLE 1 COMPARISON OF SOME KEY THERMAL PROPERTIES OF ACRYLIC AND EX-112 TRANSPARENCIES (DATA FROM REF. 1)

PROPERTY	ACRYLIC	EX–112
HEAT DISTORTION TEMPERATURE, 1750 KN/M^2, ^oC	106	112
LIMITING OXYGEN VALUE, % O_2	18	21
THERMAL CONDUCTIVITY, $W/CM/CM^2$, $^oC \times 10^3$	2.32	3.50
COEFF OF THERMAL EXPANSION, CM PER $^oC \times 10^{-6}$, AT 23^oC	60	42
TIME TO BURN THROUGH, SECONDS, 1.3 CM THICK SAMPLE (AMES T-3 TEST)	100	>1080

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